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IS 9986 (1999): Benzaldehyde [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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बेन्ज़लडिहाईड – विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

BENZALDEHYDE — SPECIFICATION

(First Revision)

ICS 71.080.80

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

August 1999 **Price Group 4**

Organic Chemicals (Miscellaneous) Sectional Committee, PCD 9

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1981. The Committee responsible for the preparation of this standard decided to revise it in order to meet the present need of the industry. In the revision, two grades have been introduced namely, Grade 1, Pure/FFC, and Grade 2, Technical. The Grade 1 is generally used in the manufacture of flavouring materials, perfumes and pharmaceuticals, while Grade 2 material is used as a chemical intermediate for dyes, pesticides and also solvent for oils, resins, cellulose ethers, cellulose acetates and nitrates. In this revision in addition to the titremetric estimation, GC method has also been specified for determining the purity. Requirement of chlorinated compounds have been modified.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BENZALDEHYDE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for benzaldehyde.

2 NORMATIVE REFERENCES

The following standards contain provisions which through, reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

IS No.	*Title*
266 : 1993	Specification for sulphuric acid (*third revision*)
1070 : 1992	Water reagent grade (*third revision*)
2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis
2631 : 1976	Specification for *iso*-propyl alcohol (*first revision*)
4905 : 1968	Methods for random sampling
5298 : 1983	Method for determination of distillation range and of distillation yield (*first revision*)

3 GRADES

The material shall be of the following two grades:

a) Grade 1 Pure/FFC grade — It is generally used in the manufacture of flavouring materials, perfumes and pharmaceuticals.

b) Grade 2 Technical grade — It is generally used in the manufacture of dyes and pesticides.

4 REQUIREMENTS

4.1 The material shall be a colourless to pale yellow liquid having odour resembling oil of bitter almonds.

4.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Annexes A to E.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable air-tight containers, such as tins, carboys, drums and tank cars.

5.2 Marking

Each container shall be securely closed after filling and marked with the following information:

a) Name of the material;
b) Mass of the material in the container;
c) Manufacturer's name and his recognized trade-mark, if any;
d) Lot or batch number;
e) Grade; and
f) Date of manufacture.

5.2.1 *BIS Certification Marking*

The container may also be marked with the Standard Mark. The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The methods of preparing the test samples and their criteria for conformity shall be as prescribed in Annex F.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in test.

Table 1 Requirements for Benzaldehyde

(*Clauses* 4.2 *and* F-4)

Sl No.	Characteristic	Requirement		Methods of Test, Ref to	
		Grade 1	Grade 2	Annex	IS No.
(1)	(2)	(3)	(4)	(5)	(6)
i)	Purity (as Benzaldehyde), percent by mass, *Min*	98.5	98.0	A	
ii)	Relative density at 27°C/27°C	1.041 to 1.043	1.040 to 1.044	B	
iii)	Acidity (as benzoic acid), percent by mass, *Max*	0.5	1.0	C	
iv)	a) Chlorinated compound	To pass the test	—	D	
	b) Chlorinated compounds (as Cl), percent by mass, *Max*	—	0.1	E	
v)	Distillation range	The difference between the temperatures running points at which 3 and 98 percent of the volume taken have been collected shall not exceed 5°C and the range shall include the temperature 178°C	—		5298

ANNEX A

[*Clause* 4.2 *and Table* 1, *Sl No.* (i)]

DETERMINATION OF PURITY

A-1 GENERAL

Two methods have been specified for determining the purity of benzaldehyde. Method A is the titremetric method while Method B is the Gas Chromatography method. GC method can be used in routine analysis, however, in case of dispute titremetric method shall be the referee method.

A-2 METHOD A — TITREMETRIC METHOD

A-2.1 Reagents

A-2.1.1 *Standard Hydroxylamine Hydrochloride Solution (Alcoholic)*

1.0 N.

A-2.1.2 *Standard Sodium Hydroxide Solution (Alcoholic)*

1.0 N.

A-2.1.3 *Bromophenol Blue Indicator*

A-2.2 Procedure

Weigh about 1-2 g of the sample accurately in a glass-stoppered, 250 ml, flat-bottomed flask and introduce 75 ml of standard alcoholic hydroxylamine hydrochloride solution. Stopper the flask and shake vigorously. Keep it at 40°C for 35 minutes and then titrate the contents with standard alcoholic sodium hydroxide solution, using bromophenol blue as indicator. Similarly, carry out the blank test.

A-2.3 Calculation

$$\text{Purity (as benzaldehyde), percent by mass} = \frac{(V_1 - V_2) \times N \times 10.6}{M}$$

where

V_1 = volume in ml of standard alcoholic sodium hydroxide solution used in sample titration,

V_2 = volume in ml of standard alcoholic sodium hydroxide solution used for blank determination,

N = normality of standard alcoholic sodium hydroxide solution, and

M = mass in g of the material taken for test.

A-3 METHOD B — GAS CHROMATOGRAPHIC METHOD

A-3.1 Apparatus

A-3.1.1 *Gas Chromatograph*

Any gas chromatograph fitted with a flame ionization detector and capable of meeting the required operating conditions.

A-3.1.2 *Integrator*

Electronic integrator is recommended but other methods of recording and measuring peak areas may be used, provided that the sensitivity requirements are met.

A-3.1.3 *Recorder*

A strip chart recorder with a scale of at least 250 mm and a response time of not more than one second for full scale deflection.

A-3.1.4 *Packed Column*

As per the specifications given below:

Length	2 m
Material of construction	Glass/SS
Internal diameter	2 mm
Solid support	Chromosorb WHP
Mesh size	80 - 100
Liquid phase	10% SE-30/10% OV-101

A-3.2 Micro Syringe

Capacity — 10 µl.

A-3.3 Procedure

Two methods can be adopted for analysis of benzaldehyde sample.

A-3.3.1 *Method 1 (Isothermal Method)*

In this method the sample is to be analysed on GC at a fixed oven temperature in an isothermal manner and GC analysis is to be continued till all the peaks corresponding to various constituents (impurities) including main peak of benzaldehyde appears in the chromatogram.

A-3.3.1.1 *Operating conditions*

Various operating conditions shall be maintained as follows:

Injector Temperature	225 ± 5°C
Detector Temperature	225 ± 5°C
Oven Temperature	110 ± 2°C
Carrier Gas	Nitrogen
Carrier flow rate	Adjust to give a retention time for benzaldehyde between 4 min and 6 min or 30 ml/min
Fuel gases	Hydrogen and air. Flow rates for flame ionization detector shall be adjusted to the levels recommended by the instrument manufacturers
Sample Size	0.2 µl is recommended but larger sample may be used provided that satisfactory resolution is maintained

A-3.3.2 *Method 2 — (Variant Method)*

In this method sample is to be injected at a initial oven temperature of 80°C in the programming mode of oven temperature with a temperature increment of 10°C/min and analysis is to be continued up to a final oven temperature of 200°C for 5 min. All other operating parameters will be same as in **A-3.3.1**.

NOTE — The above gas chromatographic conditions are suggestive. However, other GC with different columns may be used provided standardization/calibrations are done after setting up chromatographic conditions for the required resolution.

A-3.4 Calibration

Prepare standard synthetic mixtures of benzaldehyde and representative impurities on a mass percent basis. Inject all the above samples in gas chromatograph and get resolution of all the interested peaks of impurity mixture. At the end of each run stop the integrator and get the area, percent of each impurity. Draw calibration curves for each impurity and calculate response factor of each impurity from the calibration curve.

A-3.5 Calculation

The integrator automatically calculates the area, percent of each individual impurity as follows:

$$\text{Area, percent of '}X\text{'} = \frac{A \times 100}{\text{Area of all peaks}}$$

where

A = Area of component 'X'

Area, percent of each impurity when multiplied with response factor will give the actual mass percent of the concerned impurity.

percent 'X' = Area, percent of 'X' × Response factor

Assay (as Benzaldehyde), Percent by mass = 100 − (Sum of the all impurity, percent).

ANNEX B

[*Clause* 4.2 and *Table* 1, *Sl No.* (ii)]

DETERMINATION OF RELATIVE DENSITY

B-1 APPARATUS

B-1.1 Pyknometer or Relative Density Bottle

25 ml capacity.

B-1.2 Water-Bath

maintained at 27 ± 0.2°C.

B-2 PROCEDURE

Clean and dry the pyknometer or relative density bottle. Weigh it, fill it with freshly boiled distilled water, place it in the bath maintained at 27 ± 0.2°C, and allow sufficient time (about 45 min) to attain the temperature of the bath. Then insert the capillary stopper which has also been brought to 27 ± 2°C. Wipe the excess liquid from the stopper, remove the pyknometer or the relative density bottle from the bath, bring to room temperature and weigh. Empty the pyknometer or the relative density bottle, clean and dry it, and repeat the operation with the material at 27 ± 0.2°C.

B-3 CALCULATION

$$\text{Relative density at } 27°\text{C}/27°\text{C} = \frac{A-B}{C-B}$$

where

A = mass in g of the pyknometer or relative density bottle filled with the material;

B = mass in g of the dry pyknometer or relative density bottle; and

C = mass in g of the pyknometer or relative density bottle filled with water.

ANNEX C

[*Clause* 4.2 and *Table* 1, *Sl No.* (iii)]

DETERMINATION OF ACIDITY

C-1 REAGENTS

C-1.1 Neutralized Ethanol/Methanol Solution

C-1.2 Standard Alcoholic KOH Solution — 0.1 N.

C-1.3 Phenolphthalein Indicator

C-2 PROCEDURE

Take about 50 ml of neutralized ethanol/methanol solution and dissolve a 10 g sample (accurately weighed) in it. Titrate against alcoholic potassium hydroxide solution using phenolphthalein as indicator.

C-3 CALCULATION

$$\text{Acidity (as benzoic acid), percent by mass} = \frac{V_1 \times N_1 \times 12.21}{M}$$

where

V_1 = volume in ml of standard alcoholic sodium hydroxide solution consumed;

N_1 = normality of standard sodium hydroxide solution; and

M = mass in g of the material taken for test.

ANNEX D

[*Clause* 4.2 and *Table* 1, *Sl No.* (iv)(a)]

TEST FOR CHLORINATED COMPOUNDS

D-1 OUTLINE OF THE METHOD

This test serves to detect the presence of halogens in many organic compounds. It consists in heating the substance in contact with pure copper oxide in the Bunsen flame; the corresponding copper halide is formed which, being volatile, imparts an intense green or bluish-green colour to the mantle of the flame.

D-2 PROCEDURE

Push one end of a 20 cm stout copper wire into a cork (this will serve as a holder). At the other end make two or three turns and wind around a thin glass rod. Heat the coil in the outer mantle of a Bunsen flame until it ceases to impart any colour to the flame. Allow the, wire to cool somewhat and, while still warm, dip the coil into a small portion of the substance to be tested and heat again in the non-luminous flame. If the compound contains a halogen element, a green or bluish-green flame will be observed (usually after the initial smoky flame has disappeared). Before using the wire for another compound, heat it until the material from the previous test has been destroyed and the flame is not coloured.

ANNEX E

[*Clause* 4.2 and *Table* 1, *Sl No.* (iv)(b)]

DETERMINATION OF CHLORINATED COMPOUNDS

E-1 OUTLINE OF THE METHOD

The material dissolved in *iso*-propyl alcohol is treated with metallic sodium and refluxed. Under the conditions the organically bound chlorine gets converted to ionisable sodium chloride. The contents are taken up with water, acidified and treated with known volume of silver nitrate solution. The total chlorine is then determined from the amount of silver nitrate solution used up by titrating the excess silver nitrate solution against standard ammonium thiocyanate solution using ferric alum solution as indicator.

E-2 REAGENTS

E-2.1 *iso*-propyl Alcohol

conforming to IS 2631.

E-2.2 Aqueous *iso*-propyl Alcohol

50 percent (*v/v*).

E-2.3 Metallic Sodium

pure, in the form of ribbon or cut in small pieces.

E-2.4 Dilute Nitric Acid

1 : 3 (*v/v*).

E-2.5 Standard Silver Nitrate Solution

0.1 N. (*see* IS 2316).

E-2.6 Ferric Alum Indicator Solution

Saturate or dissolve 8 g of ferric ammonium sulphate in 400 ml of water containing 3 to 5 ml of concentrated sulphuric acid (*see* IS 266).

E-2.7 Nitrobenzene

E-2.8 Standard Ammonium Thiocyanate Solution

0.1 N (*see* IS 2316).

E-2.9 Standard Ethanolic Sodium Hydroxide Solution

approximately 0.5 N. Free from chlorine.

E-3 PROCEDURE

Take 50 ml of *iso*-propyl alcohol in a 250 ml flask and add 2 to 3 g of material accurately weighed. Add 3 g of metallic sodium slowly, till there is no reaction. Connect the flask to a reflux condenser and boil vigorously for 1 hour, shaking the flask occasionally, dissolve the excess metallic sodium by cautiously adding 10 ml of aqueous *iso*-propyl alcohol through the condenser at the rate of one or two drops per second. Boil for additional 10 min and then add about 50 ml of water. Cool to room temperature and disconnect the flask from condenser. Acidify the contents with dilute nitric acid until just acidic (blue litmus paper turning red). Add exactly 50 ml of silver nitrate solution, followed by 50 ml of nitrobenzene (*see* Note 1) and 2 to 3 ml of ferric alum indicator solution.

Add standard ammonium thiocyanate solution slowly from the burette to the contents in the flask, constantly swirling. At the first appearance of red colour, stopper the flask tightly and shake vigorously for 15 seconds to coagulate and remove any precipitate from aqueous phase (*see* Note 2).

Continue the titration slowly until the end point of brick red colour of aqueous phase is reached. Carry out a blank determination simultaneously following exactly the procedure used for the material.

NOTES

1 Nitrobenzene is very hazardous when absorbed through skin or when its vapour is inhaled. Such exposure may cause anemia. Do not get nitrobenzene in the eyes, on the skin or on clothing. Avoid breathing its vapour. Use only with adequate ventilation.

2 Care should be exercised in opening the flask after shaking. Pressure may develop causing small amounts of acid solution to be sprayed from the mouth of the flask as the stopper is removed.

E-4 CALCULATION

$$\text{Total chlorine (as Cl), percent by mass} = \frac{(V_1 - V_2) \times N \times 3.546}{M}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution used in the blank determination;

V_2 = volume in ml of standard ammonium thiocyanate solution used in the test with the material;

N = normality of standard ammonium thiocyanate solution; and

M = mass in g of the material taken for the test.

ANNEX F

(*Clauses* 4.2 *and* 6)

SAMPLING OF BENZALDEHYDE, TECHNICAL

F-1 GENERAL REQUIREMENTS FOR SAMPLING

F-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

F-1.2 The sampling instrument shall be clean and dry.

F-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

F-1.4 To draw representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

F-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

F-1.6 The sample containers shall be of such a size that an ullage of about 5 percent is left after pouring in the sample.

F-1.7 Each sample container shall be sealed air-tight after filling, and marked with the full details of sampling, the date of sampling and details given under **5.2**.

F-2 SCALE OF SAMPLING

F-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

F-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

F-2.3 In order to ensure the randomness of selection, random sampling procedure given in IS 4905 may be followed.

F-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

F-3.1 From each of the containers selected according to **F-2.3**, a representative portion of the material, about 300 ml in volume, shall be drawn. These samples shall constitute individual samples.

F-3.2 From each of these individual portions (**F-3.1**), an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 600 ml in volume. The composite sample shall be divided into three equal parts, one for the purchaser, one for the supplier and the third for the referee.

F-3.3 Referee Sample

The referee sample consists of the composite sample marked for this purpose, and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the two and shall be used in case of any dispute between the two.

Table 2 Number of Containers to be Selected from Lots of Different Sizes

(*Clause* F-2.2)

Sl No. (1)	Lot Size (*N*) (2)	Sample Size (*n*) (3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 200	6
v)	201 to 300	7
vi)	301 to 500	8
vii)	501 to 800	9
viii)	801 and above	10

F-4 NUMBER OF TESTS

Tests for the determination of all the characteristics given in Table 1 shall be carried out on the composite sample.

F-5 CRITERIA FOR CONFORMITY

For ensuring the conformity of the lot to the requirements of all the characteristics, the test results on the composite sample shall meet the corresponding requirements specified.

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